

Abstract No. Frat373

Study of the Effects of Additives on Microstructure of Curing Cement Paste by In Situ Synchrotron SAXS/WAXD

E.Fratini, P.Baglioni, D.Berti (CSGI, Univ. of Florence, Italy) B.S.Hsiao (SUNY, Stony Brook) and S.H.Chen (MIT) Beamline(s): X27C

Introduction: Cement is one of the most used materials in human activities. The main components of anhydrous Portland cement are alite (C_3S , about 50-70%), belite (C_2S , about 15-30%), aluminate phase (C_3A , about 5-10%), and ferrite phase (C_4AF , about 5-15%), where according to the chemical nomenclature: C=CaO, S=SiO₂, H=H₂O, A=Al₂O₃, and F=Fe₂O₃. It is generally recognized that setting and hardening of the cement are due to calcium silicate hydrate (C-S-H). A great deal of information about the C-S-H microstructure has been obtained and refined during the last decade [1-3]. In order to improve workability and increase the cement' strength by decreasing its porosity, chemical substances (superplasticizers (SP), usually polymers) are usually added in most of applications. Here, we are interested in characterizing how SP interacts with C-S-H, how they affect cement hydration, where they distribute during the setting process and how they affect the globules size and interaction.

Methods and Materials: Simultaneous SAXS and WAXD experiments were carried out at the synchrotron X-ray Beam-line X27C at the National Synchrotron Light Source in Brookhaven National Laboratory. The wavelength of the X-ray beam was 0.1336 nm. Cement hydration was followed at room temperature. The pastes had a w/c ratio of 0.5 and a 0.5% additive concentration (w/w of C_3S dry powder). Three different additives were studied: two polynaphthalenesulfonate derivatives (labeled NSF and NSF47) and a polyacrylic derivative (HSP).

Results: Figure shows the WAXD spectra in the case of C_3S powder (C_3S), C_3S after 18 days of hydration (H_2O) and after 18 of hydration in presence of SP (**NSF**, **NSF47** and **HSP**). Each peak reported is due to one or more faces in the crystalline structure. Since the data are not in absolute intensity, only a qualitative comparison can be made. The data clearly indicate that additives stop the C-S-H formation by modulating the lattice face growth. In fact, the peak at $2\theta=16$ increases less in the presence of additives. Furthermore, the peak at about $2\theta=13$ is still visible in the spectra of samples containing an additive. Peaks at $2\theta=26$, 29 and 31 are characteristic for unreacted C_3S . As we can see in terms of relative intensities, the faces corresponding to peaks 29 and 31 are more reactive when no additive is added. Moreover, NSF, NSF47 and HSP, strongly block reactions occurring at the lattice face corresponding to the peak at $2\theta=31$. These results clearly suggest that the additive have a selective binding to preferential crystal lattice directions of C_3S . The effect on the other phases (C_2S , C_3A and C_4AF) is weaker suggesting that additives mainly affect C_3S .

Conclusions: A selective effect of SP additives on the main component of a cement paste is shown.

Acknowledgments: EF, PB and DB acknowledge financial support from CSGI and ItalCementi. Authors gratefully thank Dr. L. Cassar (ItalCementi-CTG) for providing pure C_3S , C_2S , C_3A and C_4AF .

References:

- 1] Allen, A.J., et al.; *Philos. Mag. B* **56**, (1987) 263-288.
- 2] Kriechbaum, M., et al.; *Progr. Colloid Polym. Sci.* **79**, (1989) 101-105.
- 3] Jennings, H.M., *Cem. Concr. Res.* **30**, (2000) 101-116.

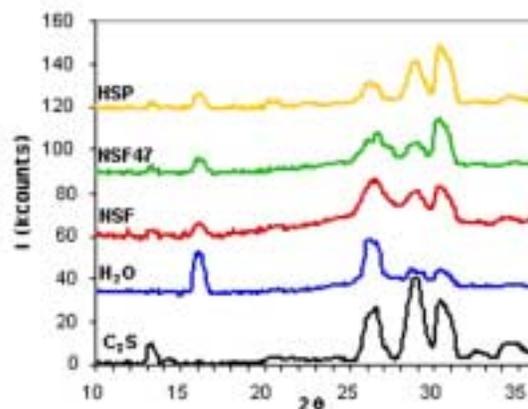


Fig.1 WAXD spectra of different cement pastes: C_3S dry powder (C_3S), hydrated (0.5 w/c) C_3S after 18 days (H_2O), hydrated (0.5 w/c) C_3S after 18 days in presence of 0.5% of different SP (**NSF**, **NSF47**, and **HSP**).